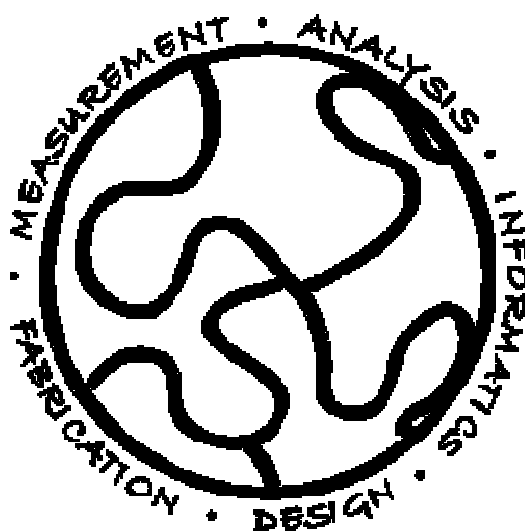


Temperature Gradient Stage

Specifications and Operation Guidelines

For applying and maintaining a temperature gradient as an orthogonal gradient in combinatorial library screening.



NIST Combinatorial
Methods Center

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1. Purpose and structure of this document

This document is provided by the NIST Combinatorial Methods Center as a guide for constructing and operating an instrument for applying and maintaining a temperature gradient. First, the basic principles of this instrument will be described. Next, the components of the instrument and schemes for its construction are supplied. Here, the discussion is based upon the specific components and design of the NCMC device (see disclaimer, page 1). Next, guidelines for operating the instrument and some basic applications are outlined, including notes on calibration and stability.

2. Principles of the gradient temperature stage.

The governing principle of the gradient temperature stage is simple: apply heat to one end of a thermally conductive material and provide a heat sink on the opposite end. As heat flows from the hot to cold, a gradient in temperature is achieved. Critical design parameters for a temperature gradient stage include:

- size (width, length, thickness) of gradient stage
- thermal conductivity of stage material
- transparency, if needed, of stage material
- size, surface area and power of heating source
- surface area of heat sink
- roughness of stage material
- ability to query the surface temperature of stage
- thermal isolation of stage from other equipment

At NCMC, we typically fabricate our temperature gradient stages out of aluminum, but other materials can be used as well (e.g., copper). When optical transparency is desired, we have employed sapphire as a stage material, which, while being moderately priced, has a much higher thermal conductivity than glass. Table 1 lists typical thermal conductivities for a variety of materials available for constructing temperature gradient stages.

Table 1. Thermal conductivities of potential gradient hot stage materials.

Material	Thermal conductivity at 293 K ($\text{Wm}^{-1}\text{K}^{-1}$)
Glass	1.4
Stainless Steel	16
Sapphire (Al_2O_3)	35
Aluminum	216
Copper	386

Heating of one end of the gradient hot stage is achieved by cartridge heaters, available from a range of suppliers including Omega, Watlow, or Chromalox. The power of the heating cartridge should be large enough to adequately provide the appropriate heat flux but small enough to minimize temperature overshoot. Temperature overshoot can also be moderated by the use of autotuning PID process/temperature controllers. Cooling of the opposite end of the hot stage can be achieved by a temperature-regulated circulating water bath for low temperature applications or by a second cartridge heater for high temperature applications. In the latter case, the temperature gradient is maintained primarily through convective heat loss from the surfaces of the gradient hot stage, with both heating cartridges “maintaining” the two end-point temperatures.

The temperature gradient can be probed either through built-in thermocouple ports or through the use of local surface thermocouples. IR temperature probes can also be employed, but for low temperature applications (typical for polymer studies) the sampling area of an IR probe can be quite large (6 mm or larger). Thus the temperature provided by an IR probe will represent an average temperature over that sampling area. It should also be pointed out that the surface temperature of the gradient hot stage may not accurately represent the temperature of a specimen placed on top of the hot stage. The thermal conductivity of the substrate (if present) and the polymer film should be considered. For thin film applications ($h < 500$ nm) on silicon wafers, the disparity in temperature should be minimal. For thick film applications ($h >$ microns) on glass slides, the disparity in temperature could be quite large ($\Delta T = 10$ °C), though it has been our experience that the slope of the temperature gradient is still consistent with the substrate gradient.

The gradient hot stage should be thermally isolated from surrounding equipment. This serves two purposes: 1) preserves the applied temperature gradient (no external sources of a heat sink), and 2) protects valuable equipment from excessive temperature exposure. Thermal isolation can be achieved in several ways. We have successfully implemented thermal isolation with Macor, a machinable glass ceramic material ($k_{\text{Macor}} = 1.4 \text{ Wm}^{-1}\text{K}^{-1}$), as well as Teflon ($k_{\text{Teflon}} = 0.25 \text{ Wm}^{-1}\text{K}^{-1}$).

The surface of the gradient hot stage can be scribed with a grid to serve as fiduciary marks for reproducible sample placement along the temperature gradient.

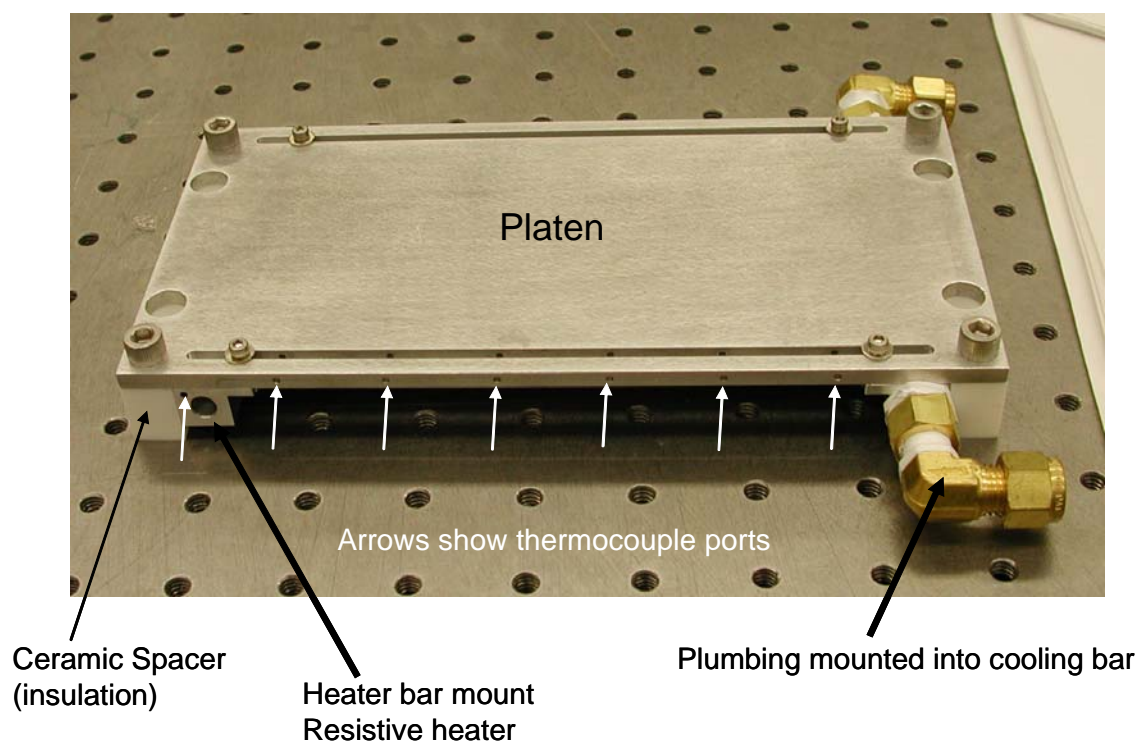


Figure 1. An NCMC gradient hot stage fabrication from aluminum. The hot side is maintained by a heating bar that accommodates a 1/4" cartridge heater. The cold side is maintained by a temperature-regulated recirculating bath plumbed using 1/8" NPT and 1/4" Swagelok fittings.

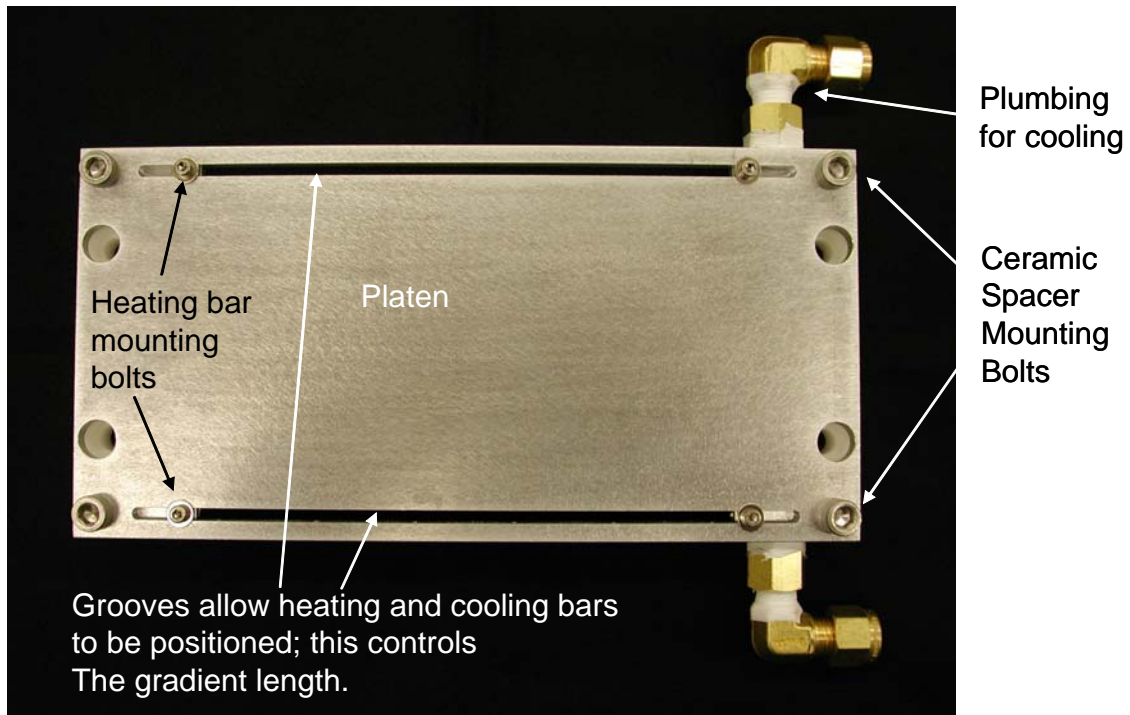


Figure 2. Plan view of a gradient hot stage illustrating the adjustable mounting brackets for the heating and cooling bars, which allows the length of the temperature gradient to be adjusted.

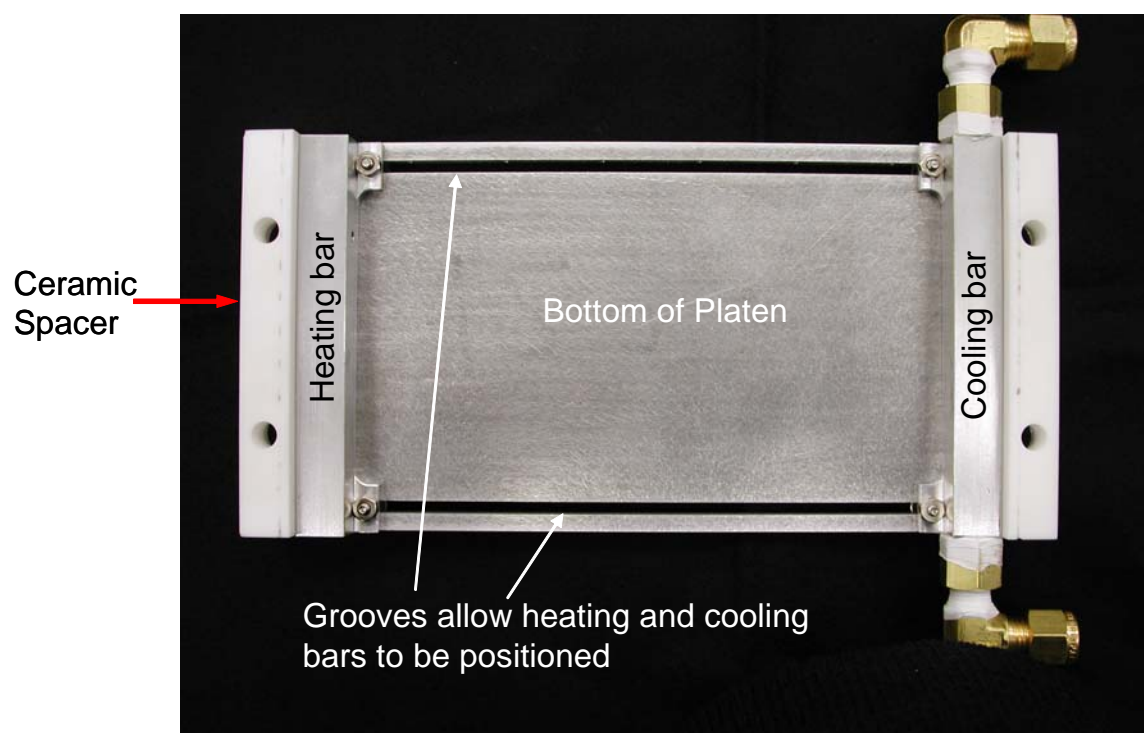


Figure 3. Bottom view of a gradient hot stage illustrating the use of ceramic spacers for thermal isolation.

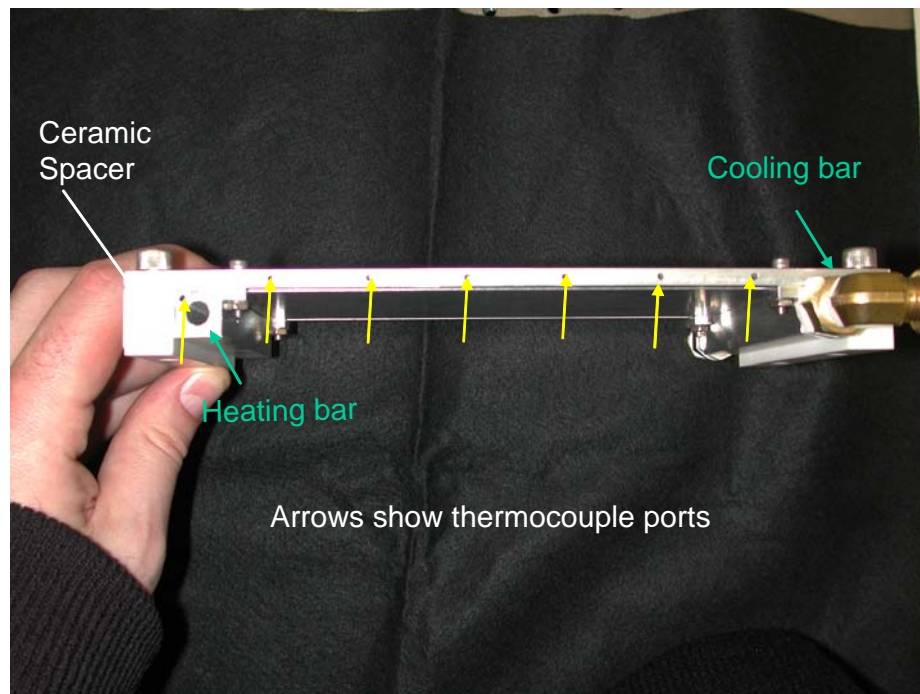


Figure 4. Bottom view of a gradient hot stage illustrating the use of ceramic spacers for thermal isolation and the use of thermocouple ports for querying the temperature gradient.

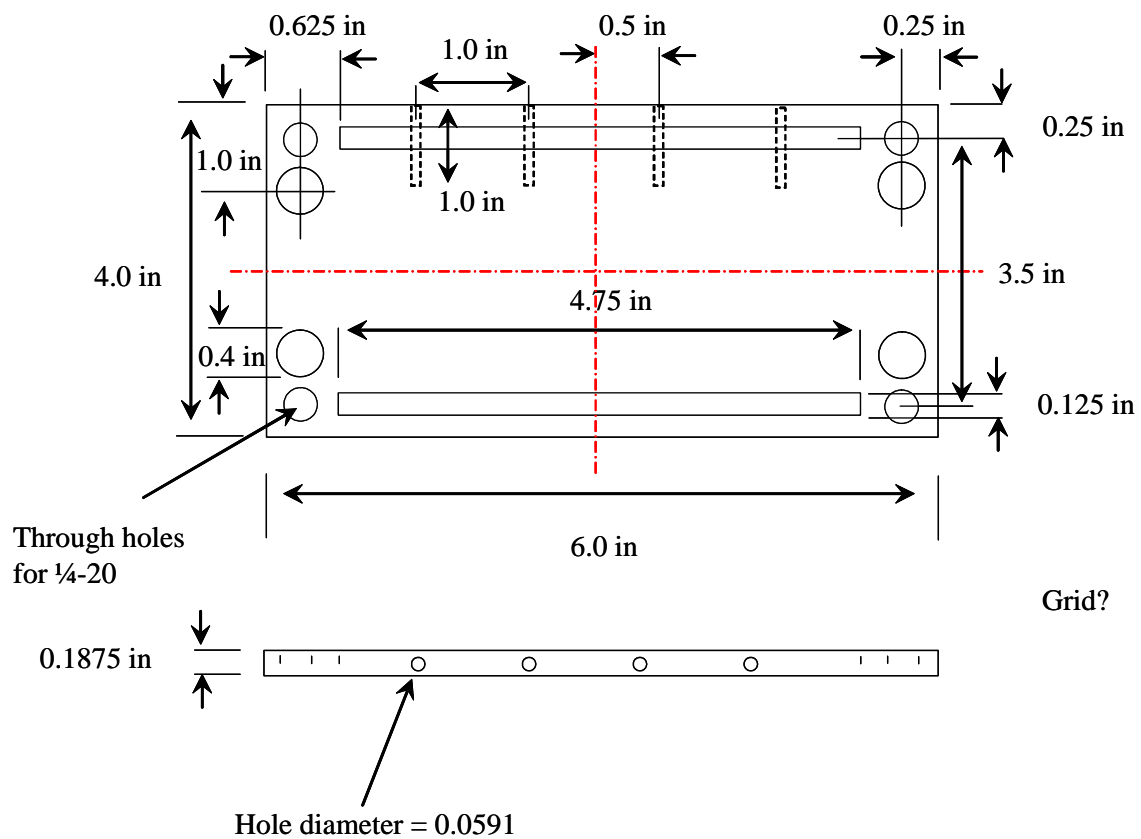


Figure 5. Technical drawings of an NCMC gradient hot stage with dimensions (4"x 6"x 3/16").

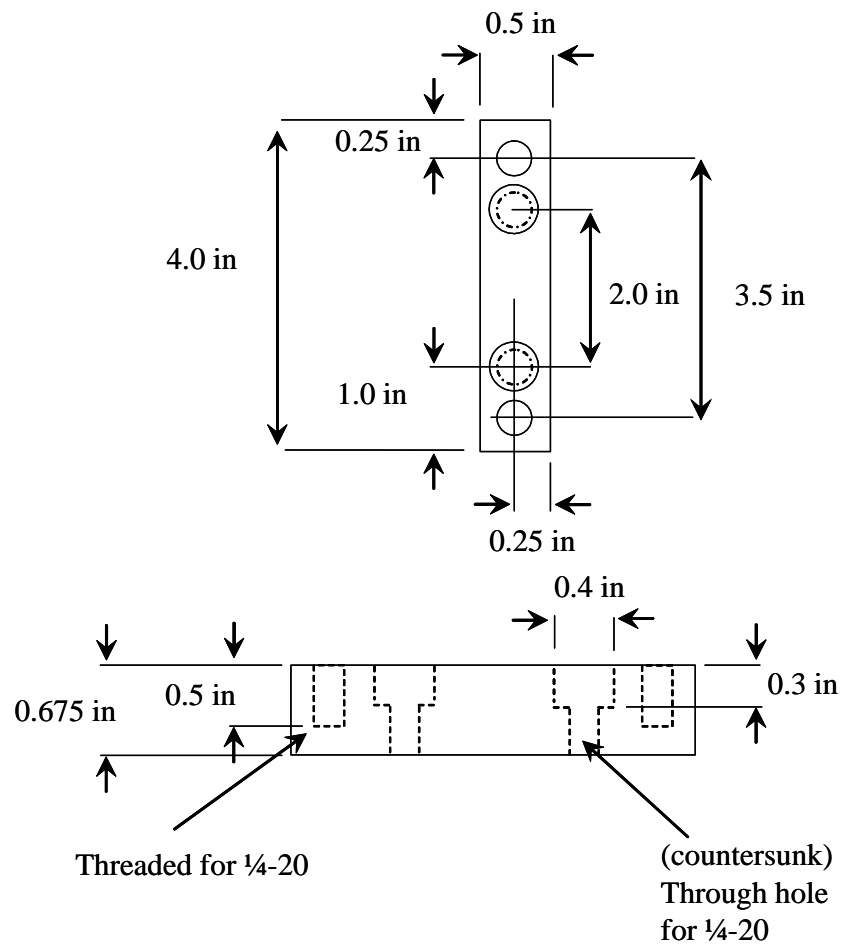


Figure 7. Technical drawing of mounting blocks used for thermal isolation. These have been fabricated in both Macor ceramic and Teflon.

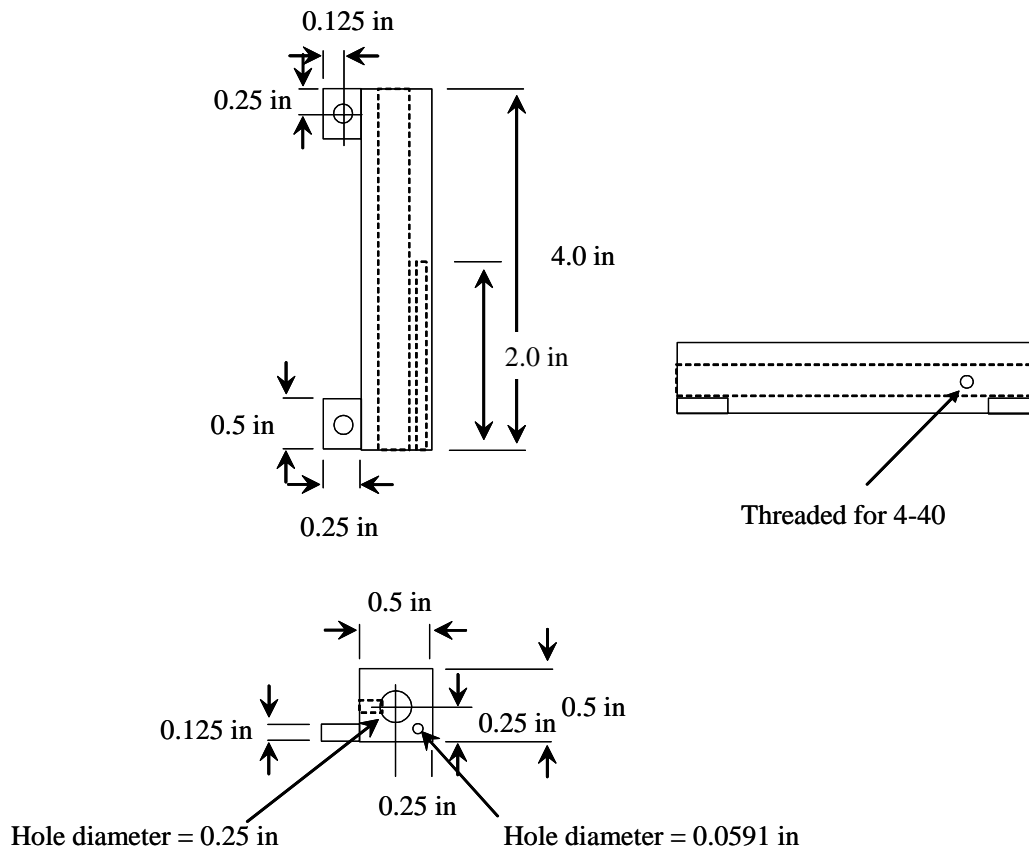


Figure 8. Technical drawing of the heating bar. The bar is designed to accommodate a 1/4" cartridge heater. It also included a thermocouple port for temperature feedback to a PID process/temperature controller.

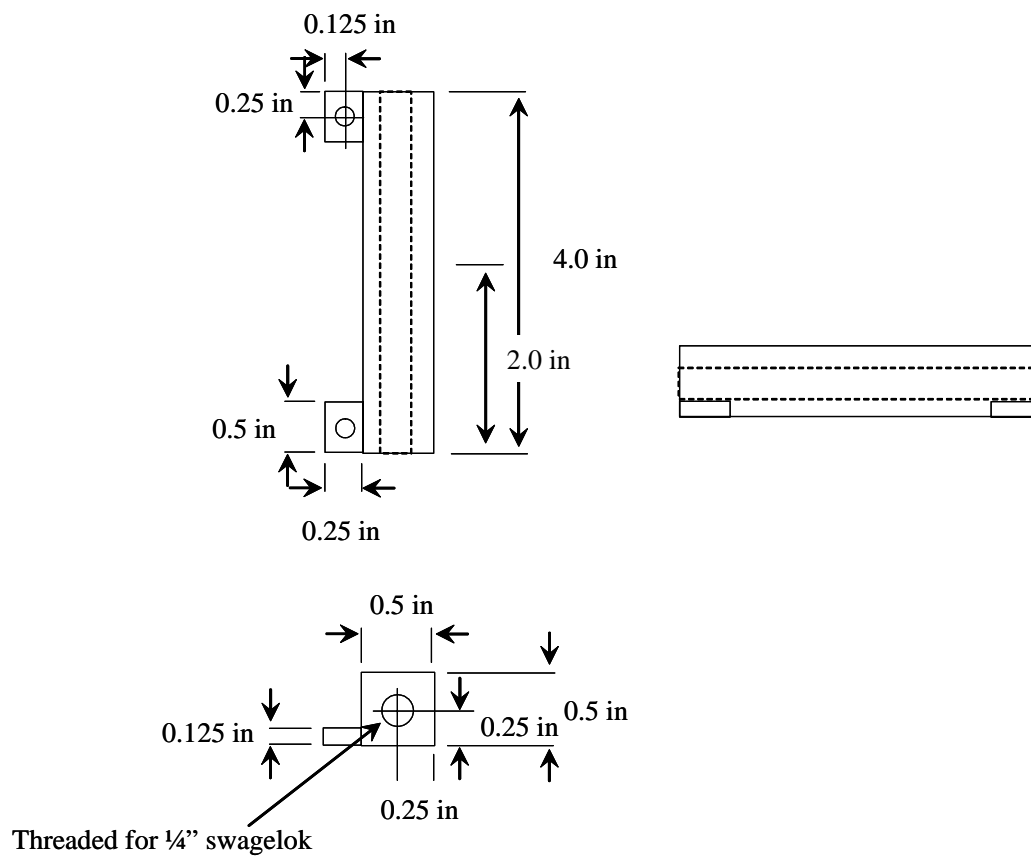


Figure 9. Technical drawing of the cooling bar. The bar has been reamed to 1/4" ID and threaded for 1/8" NPT fittings.

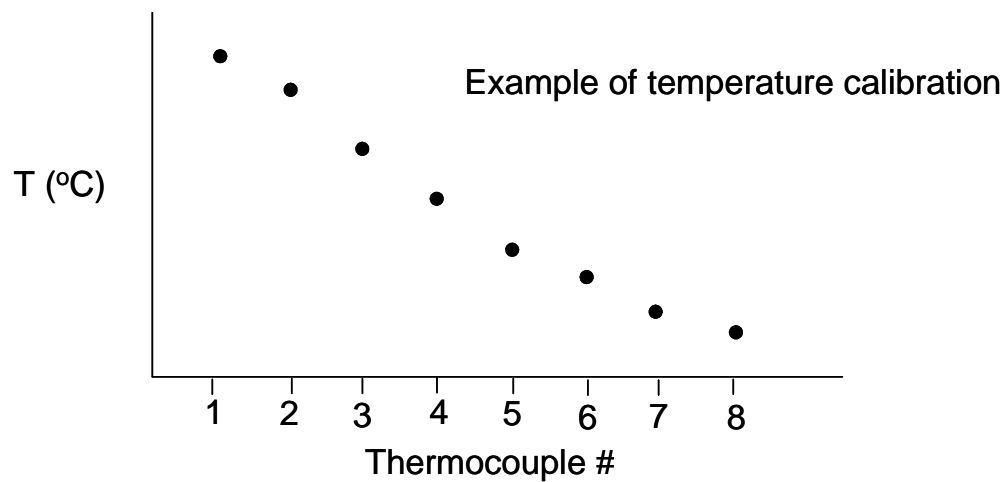
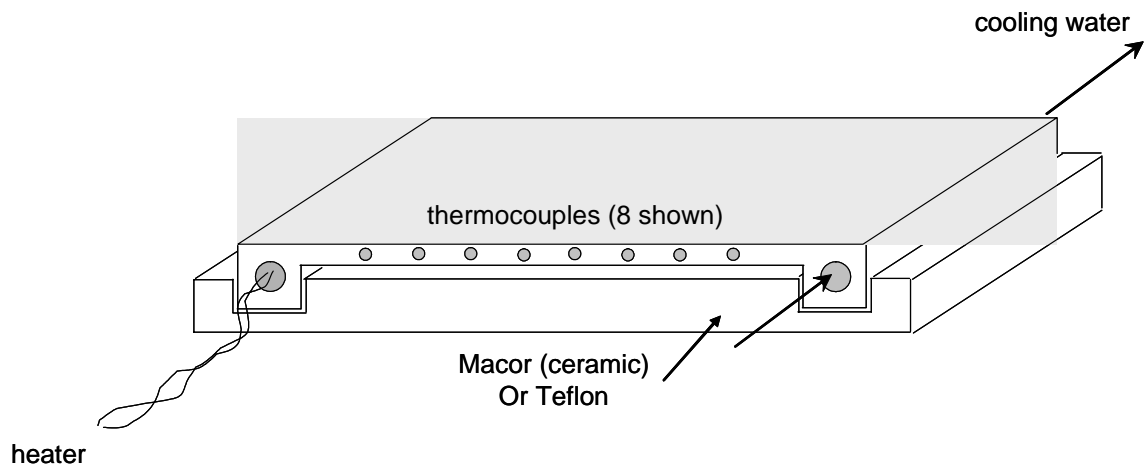


Figure 10. (Top) Schematic of a gradient hot stage supported on a base for thermal isolation. (Bottom) Example of a temperature calibration using the imbedded thermocouples along the gradient axis of the hot stage.